

## **Lower Willamette Group**

**Co-Chairperson: Bob Wyatt, NW Natural**  
**Co-Chairperson: Jim McKenna, Port of Portland**  
**Treasurer: Larry Patterson, ATOFINA**

October 1, 2004

Chip Humphrey and Eric Blischke  
US Environmental Protection Agency, Region 10  
811 SW 6th Avenue, 3rd Floor  
Portland, OR 97204

RE: Submittal of Revised Round 2 Quality Assurance Project Plan Addendum 1: Surface Water and Round 2A Field Sampling Plan: Surface Water Sampling

Dear Mssrs Humphrey and Blischke:

The Lower Willamette Group (LWG) is pleased to submit the revised Round 2 Quality Assurance Project Plan (QAPP) Addendum 1: Surface Water for your review and approval. The revised QAPP addendum incorporates comments received from EPA on September 22, 2004. In accordance with Mr. Blischke's September 27, 2004 e-mail to the LWG, the Round 2A Surface Water Field Sampling Plan will not be resubmitted. Instead, the LWG agrees to the following:

- 1) The low flow sampling event will take place in summer or early fall 2005;
- 2) The summer/early fall 2005 sampling event will include high volume/low detection limit sampling methods as described in the Surface Water FSP;
- 3) If practicable, a round of transition zone sampling will take place concurrently with the summer/early fall 2005 sampling event; and
- 4) The results of the Round 2A and Round 2B sediment coring program, the upcoming groundwater pilot study and the conceptual site model should be used to design this transition zone sampling program.

Sincerely,



Bob Wyatt  
Co-Chair



Jim McKenna  
Co-Chair

cc: LWG Executive Committee  
LWG Legal Committee



**Portland Harbor RI/FS**

**ROUND 2**  
**QUALITY ASSURANCE PROJECT PLAN**  
**ADDENDUM 1: SURFACE WATER**

October 4, 2004

**Prepared for:**  
The Lower Willamette Group

**Prepared by:**  
Integral Consulting, Inc.

## **SECTION A: PROJECT MANAGEMENT**

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### **A1 TITLE AND APPROVAL SHEET**

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#### **PORTLAND HARBOR RI/FS ROUND 2 SURFACE WATER**

##### Quality Assurance Plan Approvals

U.S. EPA Project Manager:	Chip Humphrey	_____	Date: _____
U.S. EPA Project QA Manager	Ginna Grepo-Grove	_____	Date: _____
CERCLA Project Coordinator:	Keith Pine	_____	Date: _____
Integral Chemistry QA Manager:	Maja Tritt	_____	Date: _____
CAS Project Manager:	Abbie Spielman	_____	Date: _____
CAS Laboratory QA Manager:	Lee Wolf	_____	Date: _____
NEA Project Manager:	Robert Wagner	_____	Date: _____
NEA Laboratory QA Manager:	William Kotas	_____	Date: _____
Axys Laboratory Project Manager:	Georgina Brooks	_____	Date: _____
Axys Laboratory QA Manager:	Dale Hoover	_____	Date: _____

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## **A2.2 LIST OF ACRONYMS**

ACG	analytical concentration goals
CAS	Columbia Analytical Services
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COI	chemicals of interest
CVAA	cold vapor atomic absorption spectrometry
DOC	dissolved organic carbon
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
ERA	ecological risk assessment
FSP	field sampling plan
GC/ECD	gas chromatography/electron capture detector
GC/FPD	gas chromatography/flame photometric detection
GC/LRMS	gas chromatography/low resolution mass spectrometry
GC/MS	gas chromatography/mass spectrometry
HHRA	human health risk assessment
HRGC/HRMS	high resolution gas chromatography/high resolution mass spectrometry
ICP	inductively coupled plasma
ICP-AES	Inductively coupled plasma – atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
ISA	initial study area
LWG	Lower Willamette Group
LWR	lower Willamette River
µg/L	micrograms per liter
µg/kg	micrograms per kilogram
MDL	method detection limit
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MID	multiple ion detection
MRL	method reporting limit
MSD	matrix spike duplicate
NEA	Northeast Analytical
PAH	polycyclic aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
PCP	pentachlorophenol
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
RI/FS	remedial investigation/feasibility study
RM	river mile
RPD	relative percent difference
SIM	selected ion monitoring
SOP	standard operating procedure
SVOC	semivolatile organic compound
TIC	tentatively identified compound
VOC	volatile organic compound

### **A3 DISTRIBUTION LIST**

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U.S. EPA Project Officer: Chip Humphrey  
U.S. EPA Oregon Operations Office: Eric Blischke  
U.S. EPA Project QA Manager: Ginna Grepogrove  
Oregon Department of Environmental Quality: Jim Anderson  
NOAA: Helen Hillman  
U.S. Fish & Wildlife Service: Ted Buerger  
Oregon Department of Human Services: Dave Stone  
Oregon Department of Fish & Wildlife: Rick Kepler  
Columbia River Inter-Tribal Fish Commission: Patti Howard  
Yakama Nation: Paul Ward  
Confederated Tribes of the Warm Springs Reservation of Oregon: Brian Cunningham  
Confederated Tribes of the Umatilla Indian Reservation: Audie Huber  
Confederated Tribes of the Siletz Indians: Tom Downey  
Nez Perce Tribe: Rick Eichstaedt  
Confederated Tribes of the Grand Ronde Community of Oregon: Rod Thompson  
Environment International: Valerie Lee  
Port of Portland: Jim McKenna  
Port of Portland: David Ashton  
Northwest Natural: Bob Wyatt  
Anchor Environmental LLC: Susan Thompson  
Integral Project Manager: Keith Pine  
Integral Field Sampling and Analysis Coordinator: Gene Revelas  
Integral Chemistry QA Manager: Maja Tritt  
CAS Project Manager: Abbie Spielman  
CAS Laboratory QA Manager: Lee Wolf  
NEA Project Manager: Robert Wagner  
NEA Laboratory QA Manager: William Kotas  
Axys Project Manager: Georgina Brooks  
Axys Laboratory QA Manager: Dale Hoover

## **A4 INTRODUCTION AND PROJECT ORGANIZATION**

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### **A4.1 Introduction**

This quality assurance project plan (QAPP) addendum describes procedures that will be used to complete the surface water investigation for Round 2 of the remedial investigation and feasibility study (RI/FS) for the Portland Harbor Superfund Site (Site) in Portland, Oregon. This addendum supplements the Round 2 QAPP (Integral and Windward 2004). The Round 2 QAPP describes procedures and requirements for the generation of data of documented acceptable quality that will be used for the RI/FS, including the ecological and human health risk assessments. This QAPP addendum addresses procedures that will be used for the surface water investigation that are not described in the Round 2 QAPP.

Supplemental information to Sections A and B of the Round 2 QAPP is provided in this QAPP addendum. Documents and records are described in Section A9 of the Round 2 QAPP and are not addressed further in this QAPP addendum.

Procedures for project assessment and oversight (Section C of the Round 2 QAPP) will be completed as described in the Round 2 QAPP, with one exception: Additional routine technical systems audits of analytical laboratories will not be required because all of the laboratories selected for this work have been audited in connection with sample analyses for Round 1 or for Round 2 sediment analyses. Laboratories will only be audited if serious problems are encountered, as described in the Round 2 QAPP.

Procedures for data validation (Section D of the Round 2 QAPP) will be completed as described in the Round 2 QAPP with one addition. The following protocol will be used for data validation in addition to protocols itemized in the Round 2 QAPP: *Guidance on the Documentation and Evaluation of Trace Metals Data Collected for Clean Water Act Compliance Monitoring (Draft)* (EPA 1996b).

The section numbers in this QAPP addendum correspond to U.S. Environmental Protection Agency (EPA) guidance for QAPP preparation (EPA 2002) and, in most cases, correspond to the Round 2 QAPP.

### **A4.2 PROJECT AND TASK ORGANIZATION**

The organizational structure for activities associated with the Round 2 investigation is provided in Section A4.2 and Figure A4-1 of the Round 2 QAPP. Contact information related to the surface water study is provided in Table A4-1 of this QAPP addendum.



## A5 PROBLEM DEFINITION AND BACKGROUND

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An overview of the Portland Harbor RI/FS is provided in Section A5.1 of the Round 2 QAPP. The objectives of the Round 2A surface water sampling program are to assess water quality conditions in the initial study area (ISA) and adjacent areas under different flow conditions, provide water quality data for use in the ecological and human health risk assessments, and provide water quality data for the assessment of recontamination potential during the FS.

The phasing and anticipated schedule of the Round 2 surface water sampling efforts are discussed in detail in the Surface Water Sampling Field Sampling Plan (FSP) (Integral 2004). In summary, samples will be collected at 23 stations in the lower Willamette River (LWR). Three types of samples will be collected, depending on the data needs for that particular station, using two different sampling methods (see FSP Table 2-2):

- **Single-Point Near-Bottom Samples.** Surface water will be collected in water less than 20 feet deep as close to 1 foot off the river bottom as practicable, and in no case more than 3 feet off the bottom, at 17 ecological risk assessment (ERA) sampling stations by either peristaltic pump or the Infiltrax 300 system (XAD-2) described further below.
- **Cross-Sectional River Transect Water-Column Samples.** Composite samples from the water column will be collected at three river transects using both a peristaltic pump and the XAD-2 system. The river transect samples at RM 4, 6.3, and 11 will be spatially integrated across the entire width and depth of the channel. Details on the river transect sampling approach are provided in the Surface Water Sampling FSP (Integral 2004).
- **Single-Point Water-Column Samples.** A peristaltic pump will be used to sample the three quiescent human health risk assessment (HHRA) stations: W-14 (Willamette Cove), W-20 (Swan Island Lagoon), and W-10 (Cathedral Park). Samples of the water column will be integrated vertically from 1 foot below the water surface to within 1-3 feet of the river bottom.

Most samples will be collected using the peristaltic pump method. However, certain chemicals of interest (COIs) in the LWR are hydrophobic and found at very low concentrations in the water column, and they are frequently undetected in surface water using standard analytical methods. They have the potential to accumulate in aquatic biota at concentrations that may pose a threat to human health and piscivorous wildlife. Therefore, very low analytical method reporting limits (MRLs) are needed to detect some of these chemicals at concentrations that can be compared to water quality criteria established for aquatic biota and the human health protection of ingestion of fish. Conversely, relatively standard analytical MRLs are adequate to detect concentrations of most of these COIs for comparison to water quality criteria

established for the protection of ecological receptors and for human ingestion of water.

Collection of high volumes of water is the typical sampling approach used to achieve the very low MRLs for surface water. By passing high volumes of surface water (e.g., 1,000 liters) through XAD-2 resin columns, these very dilute COIs will be concentrated as they adsorb to the resin beads. Subsequently, the target compounds can be extracted from the resin and then analyzed.

EPA has identified two surface water sample locations [W-13 (Willamette Cove) and W-15 (Rhone Poulenc)] as well as the three river transect sample locations that will require the high-volume, XAD-2 system surface water sampling method for analysis of dioxins/furans. EPA also identified two additional surface water sample locations, W-16 (ATOFINA) and W-18 (Portland Shipyard), that require high-volume sampling for analysis of pesticides and polychlorinated biphenyls (PCBs) (see FSP Figure 2-1 and Table 2-2).

All high-volume water samples collected with an XAD-2 column will be filtered. The associated pumping system has an inline filter placed before water passes through the XAD-2 resin column. The filter will be extracted and analyzed separately from the XAD-2 column extract to determine the concentrations of COIs sorbed to entrained sediment particles. The filter and XAD-2 resin analytical results can be combined to determine the total analyte concentration in the water column.

Surface water samples will be collected at three different river flow regimes to measure seasonal variability of COI concentrations. Surface water sampling is scheduled to occur during 1) low-flow conditions in the early fall, 2) the early rainy season in mid-late fall, and 3) late winter high-flow period when amphibian egg masses are typically released.

## **A6 TASK DESCRIPTION**

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The tasks to be completed for Round 2 include sample collection, laboratory analyses, data quality evaluation, data management, and report preparation. Summaries of field and laboratory tasks and references to detailed descriptions are provided in this section. Procedures for data quality evaluation, data management, and report preparation are described in the Round 2 QAPP.

### **A6.1 Field Tasks**

Two methods of surface water collection will be used during Round 2A:

1. A peristaltic pump and Teflon™ tubing will be used to collect bulk water samples.

2. An Infiltrax 300 sampling device designed by Axys Environmental Systems, which includes a stainless-steel column packed with XAD-2 resin, will be used to collect high-volume samples.

The peristaltic pump will be used to collect samples for determination of conventional analytes, metals, butyltin compounds, chlorinated herbicides, and semivolatile organic compounds (SVOCs) at all applicable sampling stations. Samples for determination of organochlorine pesticides and PCB Aroclors will be collected using the peristaltic pump or the Infiltrax 300 system, depending on reporting limit requirements for each location. Samples for analysis of chlorinated dioxins and furans, low-level polycyclic aromatic hydrocarbons (PAHs) and phthalate esters, and PCB congeners will be collected using the Infiltrax 300 system. Details regarding the sampling program and sample collection procedures are provided in the Surface Water Sampling FSP. Sample collection procedures are summarized below in Section B2.

#### **A6.2 Laboratory Analyses and Deliverables**

Chemical analysis of bulk and high-volume surface water samples will be completed by three laboratories:

- Northeast Analytical (NEA; Schenectady, NY) will complete analyses for pesticides in bulk water samples and for PCB Aroclors in bulk water samples and in XAD extracts.
- Axys Analytical (Axys; Sidney, BC, Canada) will prepare XAD extracts and will complete analyses for pesticides, PAHs, phthalate esters, chlorinated dioxins and furans, and PCB congeners in XAD extracts.
- Columbia Analytical Services (CAS; Kelso, WA) will complete all other analyses on bulk water samples (i.e., conventional analyses, metals, butyltin compounds, chlorinated herbicides, and SVOCs).

Laboratories and analyses for surface water samples are provided in Table A6-1. A complete analyte list with analytical concentration goals (ACGs) and method reporting limits (MRLs) is provided in Table A6-2.

Analyses will be completed using EPA methods and other established methods as indicated in Table A6-1. Laboratory data deliverables are described in the Round 2 QAPP.

#### **A6.3 Project Schedule**

Round 2 surface water sampling will be initiated following EPA's approval of the Surface Water Sampling FSP and this QAPP addendum. It is anticipated that Round 2 surface water sampling will take place in fall 2004 and late winter 2005.

Schedules for delivery of data and reports are described in the Programmatic Work Plan (Integral et al. 2004).

## **A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA**

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### **A7.1 The Data Quality Objective (DQO) Process**

DQOs for the surface water study are described in the Round 2 QAPP. ACGs have been established to identify analytical sensitivity levels that will be sufficient to determine risks to ecological and human health. Although ACGs can be met for many analytes, modifications to optimize laboratory MRLs are not sufficient to meet the ACGs in all cases. MRLs and ACGs for Round 2A surface water are provided in Table A6-2.

### **A7.2 Data Quality Indicators**

The overall quality objective for Round 2 is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. The QA procedures and measurements that will be used for this project are based on EPA guidance (EPA 1983, 1994b, 1999, 2004) and on established laboratory methods from other sources (Unger et al. 1986; APHA 1997).

QC samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Table A6-1. All QC requirements will be completed by each laboratory as described in the protocols and in the Round 2 QAPP. Laboratory control limits for quality control samples and procedures are provided in Tables A7.1 and A7.2 and in the laboratory QA manuals (Appendix C of the Round 2 QAPP and Appendix A of this QAPP addendum). Data validation criteria and procedures are described in Sections D1 and D2 of the Round 2 QAPP. During data validation, low recoveries will be evaluated for analytes with wide laboratory control limits to evaluate potential bias of the data. Data will be qualified on this basis if a bias is identified. PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are described in the Round 2 QAPP.

Target MRLs for this study are summarized in Table A6-2. Laboratory methods are described in Section B4. Method detection limits (MDLs) will be determined by each laboratory for each analyte, as described in the Round 2 QAPP. When available, MDLs are provided in Table A6-2. MDL studies will be completed for all remaining analyses and provided to EPA before project samples are analyzed.

Analyte concentrations for this investigation will be reported to the MDL, as described in the Round 2 QAPP. For analyses completed by Axys (i.e., pesticides, PAHs, phthalate esters, chlorinated dioxins and furans, and PCB congeners in high-volume samples), sample-specific detection limits will be reported as described in EPA methods 1613B and 1668A.

## **A8 SPECIAL TRAINING/CERTIFICATION**

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In addition to requirements for experience and training provided in Section A8 of the Round 2 QAPP, requirements for surface water sampling include experience with water sample collection for low-level analytes and training on the use of the Infiltrax 300 system. Ian Stupakoff, the field coordinator for surface water sampling, and Susan Fitzgerald have the requisite experience in collection of water samples for low-level constituents, including the use of EPA Method 1669. Mr. Stupakoff will conduct or supervise all surface water sample collection. Ms. Fitzgerald will be available if needed. Axys will provide the requisite training on the use of their sampling system prior to the first surface water sampling event.

## **SECTION B: DATA GENERATION AND ACQUISITION**

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Section B of this Round 2 QAPP addendum includes the following supplemental sections:

- B1 Sampling Process Design
- B2 Sampling Methods
- B3 Sample Handling and Custody
- B4 Field and Laboratory Methods
- B5 Quality Control.

Procedures related only to the instrumentation that will be used for field measurements of water quality conditions are additionally addressed in the following sections, while laboratory instrumentation is discussed in the Round 2 QAPP:

- B6 Instrument/Equipment Testing, Inspection, and Maintenance
- B7 Instrument/Equipment Calibration and Frequency.

The following information is provided in the Round 2 QAPP and is not addressed further in this QAPP addendum:

- B8 Inspection/Acceptance of Supplies and Consumables
- B9 Non-direct Measurements
- B10 Data Management.

Details regarding field documentation for surface water sampling are provided in the Surface Water Sampling FSP.

### **B1 SAMPLING PROCESS DESIGN**

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Composite water samples will be collected at 23 stations during each of three surface water sampling events that represent low-flow conditions (late summer or early fall), the early rainy season (fall), and high-flow conditions (winter). The 23 stations include:

- Three cross-sectional transects at the upstream (RM 11), middle (RM 6.3), and downstream (RM 4) boundaries of the ISA, to support site characterization
- Fourteen stations in areas providing amphibian habitat to support the ERA
- Three stations in human use contact areas to support the HHRA
- Three stations in beach areas to support the ERA.

Samples collected for the ERA and HHRA will also be used to support the site characterization and source evaluation. Details regarding the sampling design for surface water are provided in the Surface Water Sampling FSP (Integral 2004).

Field QC samples to be collected for each sampling event are summarized in Table B4-3. Replicate water samples and field splits of samples collected by peristaltic pump will be collected at approximately 5% of the water sampling stations. Equipment rinsate blanks will be collected for the peristaltic pump near the beginning and again near the end of each sampling event. Storage blanks will also be collected near the beginning and end of each sampling event. These will be analyzed if contaminants are found in the equipment blanks at levels above 20% of analyte concentrations in any sample (i.e., the analyte concentration in any sample is less than 5 times the concentration in a related equipment blank) to evaluate the source of the contamination. Prior to the start of fieldwork for each sampling event, decon blanks will be analyzed to ensure that the decontamination procedure used for the peristaltic pump equipment is adequate. The field blank for the XAD-2 system will consist of blank resin columns that will be shipped with the samples. These columns will be analyzed, if needed, to evaluate results obtained for surface water samples.

## **B2 SAMPLING METHODS**

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This section outlines sample collection methods, equipment, and sample requirements for Round 2 surface water samples. Additional details not provided here are included in Surface Water Sampling FSP (Integral 2004). Sample containers, sample size requirements, preservation, and holding times for surface water samples and equipment rinse blanks are summarized in Table B2-1. Corrective actions are addressed in Sections B2.3 and C1 of the Round 2 QAPP.

### **B2.1 Sample Collection**

Surface water samples will be collected by peristaltic pump at all locations. The Infiltrax 300 system will additionally be used to collect high-volume samples at seven locations for the low-flow and early rainy season sampling events and at three locations for the high-flow sampling event.

Surface water sampling will follow the guidelines in EPA Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA 1996a), and in the *Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001). High-volume surface water sampling will follow guidelines established in EPA's SOP MSL-M-090-00 (EPA 1994a) and in *Total Maximum Daily Loads for Dioxins in the Houston Ship Channel Quality Assurance Project Plan* (Houston and TNRCC 2002). A single composite sample will be collected at each of the three transect stations (i.e., downstream boundary of the ISA, mid-ISA, and upstream of the ISA). The composite sample will be collected in a manner that reflects the cross-sectional area and flow regime of the river transect.

Details regarding surface water sample collection are provided in the Surface Water Sampling FSP (Integral 2004).

## **B2.2 Surface Water Sampling by Peristaltic Pump**

Surface water samples for standard chemical and conventional analyses will be collected using a peristaltic pump with an extended sampling tube lowered to the desired depth. Samples will be collected using the two-person “clean hands – dirty hands” method (EPA 1996a). The peristaltic pump’s water intake will be placed 15 feet away from the bow of the boat with a long pole. The outflow of the pump will be directed through a Y-splitter into two composite mixing containers for sampling.

Equal volumes will be pumped into two large, pre-cleaned, 10-L or 20-L mixing containers equipped with magnetic stirring devices. The first container, made of polycarbonate, will be used for compositing and mixing samples for subsequent analysis of trace metals, butyltin species, and conventional analytes. The second container, made of stainless steel or glass, will be used for compositing and mixing samples for subsequent analysis of organic compounds. Following sample compositing in the mixing containers, appropriate sample bottles will be filled using a second peristaltic pump, with the outflow directed into the bottle. The sample jar will be held near the pump outlet, and the sample container will be rinsed several times and then filled. The sample containers will be capped, labeled, and placed in clean, double Ziploc™ bags, and then placed inside a cooler.

Two types of surface water samples will be collected by peristaltic pump: unfiltered and filtered [for metals and dissolved organic carbon (DOC)]. For filtered metals and DOC samples, the filter will be placed in-line near the tubing outlet to filter samples immediately before the water is discharged into the sample bottle. A 0.45 µm filter will be used for dissolved metals samples. The filter size for DOC will be selected in consultation with EPA.

Samples for total suspended solids (TSS) and total dissolved solids (TDS) will be filtered at the laboratory. The filter size to be used for these analyses will be determined by EPA and LWG.

## **B2.3 High-Volume Surface Water Sampling by XAD-2 Columns**

High-volume surface sampling will be accomplished using an Infiltrax 300 system with XAD-2 resin columns. This sampling method will be used to collect hydrophobic organic compounds from water at locations that require ultra-low analytical detection methods.

Approximately 1,000 L of water will be pumped through Teflon™ tubing, glass fiber filter cartridges, and Amberlite XAD-2 resin beads packed inside stainless-steel canisters. Suspended particles will be retained on the filters and dissolved organic contaminants will be adsorbed onto the resin. This method eliminates the need to collect, store, and transport large volumes of water. The minimum filter pore size to



be used will be determined in consultation with EPA and its partners before sampling is initiated.

Samples will be collected using the “clean hands – dirty hands” method. Once the desired volume is pumped, the column assembly will be removed and any residual water will be drained out. XAD-2 canisters will be labeled, wrapped appropriately, and then placed in a cooler with wet ice. Any additional glass fiber filters will be removed, placed in glass jars, and stored in a cooler containing dry ice.

At the analytical laboratory, the column and filters will be analyzed individually to determine the dissolved and particulate concentrations of analytes in the samples. The sum of the dissolved and particulate concentrations will represent the total analyte concentration in the surface water samples.

### **B3 SAMPLE HANDLING AND CUSTODY**

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Sample custody procedures are described in Section B3 of the Round 2 QAPP. Sample preservation and storage requirements and holding times for the surface water samples are provided in Table B2-2 of this QAPP addendum.

Samples collected by the Infiltrex 300 system will be analyzed for PCB Aroclors. In order to allow possible future analysis of these samples for PCB congeners, a portion of each sample extract will be stored frozen at Axys. Portions of extracts from both the XAD-2 resin (for dissolved PCBs) and the filters (for PCBs bound to particulates) will be archived in this manner.

### **B4 FIELD AND LABORATORY METHODS**

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#### **B4.1 Field Measurements**

*In situ* measurements of general water quality characteristics will be taken at all sampling stations, including conductivity, pH, temperature, dissolved oxygen, and oxidation-reduction potential. Measurements will be taken using a YSI 650/600XLM Multi Probe lowered into the water column. Details regarding the operation of this instrument, including calibration, measurement, quality control procedures, and decontamination, are provided in the *YSI 650/600XLM Multi Probe SOP*, provided as Appendix E of the Surface Water Sampling FSP.

#### **B4.2 Laboratory Analyses**

Laboratory methods to be used for Round 2 are consistent with requirements provided in EPA methods (EPA 1983, 1994b, 1999, 2004) and other widely accepted protocols (Unger et al. 1986). Modifications will be made to these methods, as necessary and technically feasible, to improve MRLs. Methods and modifications are described below. Analytes, MRLs, and ACGs for sediment samples are provided in Table A6-2. Method modifications will not be sufficient to reduce MRLs to the level of the

ACGs for several analytes, and ACGs will not be attained in these cases. Laboratory methods are described below for surface water samples.

Surface water samples collected by peristaltic pump will be analyzed for the following:

- Conventional analyses
- Total and dissolved metals
- Butyltin compounds
- Chlorinated herbicides
- Organochlorine pesticides
- PCB Aroclors
- SVOCs.

Surface water samples collected by the Infiltrax 300 system will be analyzed for the following:

- Organochlorine pesticides
- PCB Aroclors
- PAHs
- Phthalate esters
- Chlorinated dioxins and furans
- PCB congeners (extract archived for potential analysis).

The total number of samples and the analyses that will be conducted on each sample are indicated in the Surface Water Sampling FSP. The laboratory methods for sample preparation and analysis are summarized in Table A6-1. Samples to be analyzed for PCB congeners and the suite of congeners to be analyzed will be selected by EPA and LWG after data for PCB Aroclors are available.

#### **B4.2.1 Conventional Analyses in Surface Water**

Conventional analyses of surface water samples will include total suspended solids, total dissolved solids, total organic carbon, dissolved organic carbon, and perchlorate. EPA methods will be used as shown in Table A6-1.

TSS and TDS will be determined gravimetrically according to EPA Methods 160.2 and 160.1, respectively. The filter pore size to be used at the laboratory for these analyses will be determined by EPA and LWG.

Total and dissolved organic carbon will be analyzed by EPA Method 415.1. Organic carbon in the samples will be oxidized and the evolved CO<sub>2</sub> will be analyzed using an infrared detector. Samples will be pretreated with hydrochloric acid to remove inorganic carbon.

Perchlorate will be analyzed according to EPA Method 314.0. Samples will be filtered at the laboratory as specified in the method and analyzed by ion chromatography.

#### **B4.2.2 Metals and Butyltin Compounds in Surface Water**

Three methods will be used to analyze surface water samples for total and dissolved metals (Table A6-1). Digestion with nitric and hydrochloric acids will be used to prepare samples for analysis of metals other than mercury and hexavalent chromium. Analysis for these metals will be completed by inductively coupled plasma/mass spectrometry (ICP/MS). Samples for analysis of arsenic will be concentrated by a factor of 10 prior to analysis as described above.

Mercury samples will be extracted with aqua regia and oxidized using potassium permanganate. Analysis will be completed by cold vapor atomic absorption spectrometry (CVAA).

Analysis for hexavalent chromium will be completed by coprecipitation of the hexavalent chromium as lead chromate with lead sulfate in a solution of acetic acid. The chromium-containing precipitate will be washed to remove trivalent chromium in solution. The precipitate will be redissolved and analyzed by ICP-AES.

The procedure for butyltins will include extraction, derivitization, and analysis of mono-, di-, tri-, and tetrabutyltin. Troponone in methylene chloride will be used to extract the butyltins, followed by a Grignard reaction with hexylmagnesium bromide to form hexyl derivatives. The extract will be cleaned up using silica and alumina cartridge columns and analyzed by gas chromatography/flame photometric detection (GC/FPD).

#### **B4.2.3 Organic Compounds in Surface Water**

##### ***B4.2.3.1 Samples Collected by Peristaltic Pump***

For analysis of chlorinated herbicides, water samples will be adjusted to a pH <2, and the herbicides in both acid and derivatized forms will be extracted with ethyl ether. Derivatives of the phenoxy acid herbicides in the extract will be hydrolyzed to the acid form by the addition of sodium hydroxide. The sample will be acidified and the acid herbicides extracted. The acids will then be converted to their methyl esters using diazomethane. Extracts will be analyzed by gas chromatography/electron capture detector (GC/ECD). Pentachlorophenol will not be included in the analyte list for this method because Method 8270C will yield a sufficiently low MRL.

Organochlorine pesticides will be extracted from samples using continuous liquid-liquid extraction procedures. Samples will be analyzed by GC/ECD. Florisil<sup>®</sup> column clean-up will be performed on the sample extracts. Pesticide detections will be confirmed at CAS by reverse search of the gas chromatography/mass spectrometry (GC/MS) data when concentrations are sufficiently high.

Solid phase extraction with an SPE-DEX 4790 extractor from Horizon Technologies and styrene divinylbenzene extraction disks will be used for PCB analyses. Four liters of sample will be extracted to provide MRLs that meet the ACGs for this project. Acid cleanup will be performed on the sample extract followed by Florisil<sup>®</sup> cleanup. Samples will be analyzed for PCB Aroclors by GC/ECD. The surrogate compounds, tetrachloro-*m*-xylene and decachlorobiphenyl, will be added to every sample and QC sample and to continuing calibration standards. NEA will complete an MDL study and initial demonstration of proficiency using the proposed method modifications prior to analysis of project samples.

Sample extractions for SVOCs will be completed using continuous liquid-liquid extraction. SVOCs will be analyzed by GC/MS with a large-volume injector to enhance sensitivity. Tentatively identified compound (TICs) will not be reported. Analyses for PAHs will be completed by GC/MS with selected ion monitoring (SIM).

#### *B4.2.3.2 Samples Collected by the Infiltrax 300 System*

Samples collected using the Infiltrax 300 system with XAD-2 resin and glass fiber filters will be extracted at Axys by Soxhlet extraction. Each extract will be divided into six portions and analyzed as follows:

- One sixth of the extract each will be used for analysis of pesticides, PCB Aroclors, PAHs, and phthalate esters
- Two sixths of the extract will be used for analysis of chlorinated dioxins and furans

Analyses for pesticides, PAHs, phthalate esters, and chlorinated dioxins and furans in the XAD-2 and glass fiber filter sample extracts will be completed by Axys. All of these analyses will be completed by GC/MS using isotope dilution methodology using multiple <sup>13</sup>C labeled surrogate standards. Analyses for PAHs and phthalate esters will be completed by GC/LRMS, while analyses for pesticides, chlorinated dioxins and furans, and PCB congeners will be completed by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

Cleanup procedures for chlorinated dioxins and furans will include sulfuric acid cleanup and silica/carbon column cleanup. Additional cleanup procedures will be used, as necessary, to remove analytical interferences. As described in EPA Method 1613B, detection limits are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer. The

detection limits listed in Table A6-2 are representative of MDLs typically obtained when using the described procedure and sample volume.

The extract for pesticide analyses will be cleaned up and fractionated by absorption column chromatography to produce two fractions. The first fraction, called E1, contains the PCBs and non-polar and moderately polar pesticides. The E1 fraction will be analyzed by HRGC/HRMS for chlorinated pesticides. The second fraction, E2, contains the most polar chlorinated pesticides and will be analyzed by HRGC/HRMS separately from fraction E1.

The extract for PAH analyses will be fractionated on silica gel into slightly polar (F1), moderately polar (F2), and highly polar (F3) fractions. The F1 fraction contains the alkane and hopane compounds. This fraction will be discarded. The F2 fraction contains PAHs. This fraction will be analyzed as described below. The third fraction (F3) contains polar hydrocarbons such as sterols. This fraction will be discarded.

Analysis will be completed by HRGC/LRMS in the multiple ion detection (MID) mode to enhance sensitivity. At least two ions will be monitored for each target compound or labeled standard. Isotope dilution quantification protocols will be used to calculate analyte concentrations.

The sample extract for phthalate esters will be fractionated on a silica gel column into non-polar and polar fractions. The polar fraction will be analyzed for phthalate esters. Analysis will be completed by HRGC/LRMS as described above for PAHs..

Analyses for PCB Aroclors in the XAD-2 extracts will be conducted at NEA by GC/ECD. Cleanup and analysis procedures will be completed as described above in Section B4.2.3.1 for samples collected by peristaltic pump.

The samples to be analyzed and the analyte list for PCB congeners will be determined by LWG and EPA data users when PCB Aroclor data are available. The E1 fraction collected during Florisil® fractionation for pesticides will be used to analyze for PCB congeners. The E1 fraction will be subjected to further cleanup procedures and analyzed by HRGC/HRMS following protocols provided in EPA Method 1668A.

## **B5 QUALITY CONTROL**

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Quality control samples will be prepared in the field and at the laboratories to monitor the bias and precision of the sample collection and analysis procedures.

Field QC samples for this study will include field replicates, field splits, equipment rinse blanks, storage blanks, decon blanks, and temperature blanks. A summary of field QC samples that will be collected for each surface water sampling event is provided in Table B4-3.

Field replicates, field splits, equipment rinse blanks, and temperature blanks are described in the Round 2 QAPP. Storage blanks are sample jars that are filled directly with the water that is used to decontaminate equipment and to collect equipment rinse blanks without passing the water over any sampling equipment. Storage blanks are kept with the samples during shipping and storage at the laboratory. Storage blanks are collected to evaluate the source of the contamination. They will only be analyzed if contaminants are found in the equipment blanks at levels above 20% of analyte concentrations in any sample (i.e., the analyte concentration in any sample is less than five times the concentration in a related equipment blank). Prior to the start of sample collection activities for each sampling event, a decon blank will be generated by the laboratory that conducts decontamination of the peristaltic pump sampling equipment. To ensure that the decontamination procedure in Appendix B is adequate, the decon blank will be analyzed by the laboratories identified in Table A6-1 for metals and organic parameters, as indicated in Table B4-3.

Laboratory quality control samples and procedures will be completed as described in the Round 2 QAPP.

## **B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE**

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Maintenance of the YSI 650/600XLM Multi Probe that will be used for field measurements will be completed as described in the manufacturer's instructions and the SOP for its use (Appendix E of the Surface Water Sampling FSP).

Testing, inspection, maintenance, setup, and calibration of laboratory instruments are described in the Round 2 QAPP.

## **B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY**

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A YSI 650/600XLM Multi Probe will be used for field measurements (i.e., conductivity, pH, temperature, dissolved oxygen, and oxidation reduction potential). This instrument will be shipped to the field pre-calibrated by Hoskin Scientific, Vancouver, BC. Calibration checks will be performed on the Multi Probe prior to use each day that measurements will be made, as described in the SOP for use of the YSI 650/600XLM Multi Probe (Appendix E of the Surface Water Sampling FSP) and in the manufacturer's instructions. Calibration checks will be completed twice daily at a minimum. The unit will be checked for calibration before each daily sampling begins and again at the end of the day. If measurements are made at more than 10 stations in a single day, calibration checks will be made after every 10 stations are measured and at the end of the day. Calibration information will be recorded in the field logbook.

Calibration procedures for laboratory instruments are described in the Round 2 QAPP.

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Table A4-1. Project Team Contact Information.

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Table A6-1. Laboratory Methods for Surface Water Samples.

Analytes	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
<i>Peristaltic Pump Samples</i>					
<b>Conventional Analyses</b>	CAS				
Total Suspended Solids		EPA 160.2	Filtration and drying	EPA 160.2	Balance
Total Dissolved Solids		EPA 160.1	Filtration and drying	EPA 160.1	Balance
Total Organic Carbon		EPA 415.1	Chemical oxidation	EPA 415.1	Infrared detector
Dissolved Organic Carbon		EPA 415.1	Filtration, chemical oxidation	EPA 415.1	Infrared detector
Perchlorate		EPA 314.0	Filtration, 0.45 um filter	EPA 314.0	Ion chromatography
<b>Metals</b>	CAS				
		EPA 3005	Acid digestion	EPA 200.8	ICP/MS
Aluminum, antimony, cadmium, total chromium, copper, lead, nickel, selenium, silver, zinc					
Arsenic		EPA 3005A (Modified)	Acid Digestion/pre-concentration	EPA 200.8	ICP/MS
Chromium, hexavalent		EPA 7195	Coprecipitation	EPA 6010B	ICP/OES
Mercury		EPA 7470	Acid digestion/oxidation	EPA 7470	CVAA
Hardness (Ca, Mg)		EPA 3005	Acid digestion	EPA 200.8 or 6010B SM2340B	ICP/MS or ICP/OES Calculation
<b>Butyltins</b>	CAS	Unger et al.	Solvent extraction Derivatization	M.A. Unger et al.	GC/FPD
<b>PCBs Aroclors</b>	NEA	EPA 3545 EPA 3640A EPA 3630C EPA 3665A	Pressurized fluid extraction Gel permeation chromatography Florisil® cleanup Sulfuric acid cleanup	EPA 8082	GC/ECD
<b>Chlorinated Herbicides</b>	CAS	EPA 8151A	Solvent extraction Esterification	EPA 8151A	GC/ECD
<b>Organochlorine Pesticides</b>	NEA	EPA 3545 EPA 3640A EPA 3660B EPA 3630C	Pressurized fluid extraction Gel permeation chromatography Sulfur cleanup (as needed) Florisil® cleanup	EPA 8081A	GC/ECD
<b>Semivolatile Organic Compounds</b>	CAS	EPA 3520C	Continuous liquid-liquid extraction	EPA 8270C	GC/MS with LVI GC/MS-SIM (PAHs only)
<i>XAD-2 Column Extracts</i>					
<b>Column Extraction</b>	Axys	EPA 3540	Soxhlet extraction	--	--
<b>PCBs Aroclors</b>	NEA	EPA 3665A EPA 3620B	Sulfuric acid cleanup Florisil® cleanup	EPA 8082	GC/ECD
<b>Organochlorine Pesticides</b>	Axys	AXYS SOP MLA-028	Florisil® cleanup Extract fractionation	AXYS SOP MLA-028	HRGC/HRMS
<b>Polycyclic Aromatic Hydrocarbons</b>	Axys	AXYS SOP MLA-021	Extract fractionation	AXYS SOP MLA-021	HRGC/LRMS
<b>Phthalate Esters</b>	Axys	AXYS SOP MLA-027	Extract fractionation	AXYS SOP MLA-027	HRGC/LRMS
<b>Chlorinated Dioxins and Furans</b>	Axys	EPA 1613B <sup>1</sup>	Layered Acid/Base/SiO <sub>3</sub> column Florisil® cleanup Carbon/Celite clean-up column 1% deactivated basic Alumina	EPA 1613B	HRGC/HRMS
<b>PCB congeners<sup>2</sup></b>	Axys	EPA 1668A	Florisil® cleanup Extract fractionation Layered Acid/Base SiO <sub>3</sub> Alumina	EPA 1668A	HRGC/HRMS

<sup>1</sup> Cleanup procedures indicated in this table will be used as needed for chlorinated dioxin and furan analyses.

<sup>2</sup> An aliquot of the XAD extracts will be archived for potential PCB congener analysis.

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
				Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
<b>Conventional Analyses, mg/L (ppm)</b>							
Total Suspended Solids	1 <sup>5</sup>	1 <sup>5</sup>	1 <sup>5</sup>	1	1		
Total Dissolved Solids	NE	NE	NE	5	5		
Total Organic Carbon	NE	NE	NE	0.07	0.5		
Dissolved Organic Carbon	NE	NE	NE	0.07	0.5		
Hardness (Ca, Mg)		NA	NA	0.2	1		
<b>Metals/Inorganics, mg/L (ppm)</b>							
Aluminum	0.087	0.087	0.087	0.0007	0.002		
Antimony	0.015	0.015	0.015	0.00002	0.00005		
Arsenic	0.000045	0.000045	0.000014	TBD	0.00005		
Cadmium <sup>6</sup>	0.000094	0.000094	0.000094	0.00001	0.00002		
Chromium, total	NE	NA	NA	0.00006	0.0002		
Chromium, hexavalent	0.002	0.002	0.002	0.003	0.010		
Copper <sup>6</sup>	0.00023	0.00023	0.00023	0.00004	0.0001		
Lead <sup>6</sup>	0.000541	0.000541	0.000541	0.00001	0.00002		
Mercury	<0.00023	<0.00023	<0.00023	0.0001	0.0002		
Nickel <sup>6</sup>	<0.005	<0.005	<0.005	0.00004	0.0002		
Selenium	0.005	0.005	0.005	0.0002	0.001		
Silver	0.00012	0.00012	0.00012	0.00001	0.00002		
Zinc <sup>6</sup>	0.03	0.03	0.03	0.0002	0.0005		
Perchlorate	0.0036	0.0036	0.0036	0.0005	0.002		
<b>Butyltins<sup>7</sup>, µg/L (ppb)</b>							
Monobutyltin	NE	NE	NE	0.0047	0.05		
Dibutyltin	NE	NE	NE	0.005	0.05		
Tributyltin	0.072	0.072	0.072	0.0071	0.02		
Tetrabutyltin	NE	NE	NE	0.0032	0.05		

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
				Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
<b>PCBs Aroclors, µg/L (ppb)</b>							
Aroclor 1016	0.230	0.000064	0.0000064	TBD	0.0025	0.00006	
Aroclor 1221	0.014	0.000064	0.0000064	TBD	0.0025	0.00006	
Aroclor 1232	0.014	0.000064	0.0000064	TBD	0.0025	0.00006	
Aroclor 1242	0.014	0.000064	0.0000064	TBD	0.0025	0.00006	
Aroclor 1248	0.0019	0.000064	0.0000064	TBD	0.00125	0.00003	
Aroclor 1254	0.0019	0.000064	0.0000064	TBD	0.00125	0.00003	
Aroclor 1260	0.014	0.000064	0.0000064	TBD	0.0025	0.00006	
Aroclor 1262	0.014	0.000064	0.0000064	TBD	0.0025	0.00006	
Aroclor 1268	0.014	0.000064	0.0000064	TBD	0.0025	0.00006	
Total PCBs	0.034	0.000064	0.0000064				
<b>Chlorinated Herbicides, µg/L (ppb)</b>							
Dalapon	1100	1100	1100	0.06	0.4		
Dicamba	1100	1100	1100	0.071	0.4		
MCPA	NE	NE	NE	24	100		
Dichlorprop	NE	NE	NE	0.061	0.4		
2,4-D	360	360	360	0.079	0.4		
2,4,5-TP (Silvex)	290	290	290	0.085	0.2		
2,4,5-T	360	360	360	0.017	0.2		
2,4-DB	290	290	290	0.13	0.4		
Dinoseb	36	36	36	0.091	0.2		
MCPP	360	360	360	23	100		
<b>Organochlorine Pesticides, µg/L (ppb)</b>							<b>pg/L (ppq)</b>
a - BHC	0.004	0.0049	0.00049	TBD	0.0005		0.6-3.0
b - BHC	0.004	0.017	0.0017	TBD	0.0005		0.6-3.0
g - BHC (Lindane)	0.052	0.052	0.0063	TBD	0.0005		0.6-3.0
d - BHC	0.004	0.004	0.004	TBD	0.0005		0.6-3.0
Heptachlor	0.0038	0.000079	0.0000079	TBD	0.0005		0.6-3.0

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
				Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
Aldrin	0.004	0.00005	0.000005	TBD	0.0005		0.6-3.0
Heptachlor epoxide	0.0038	0.000039	0.0000039	TBD	0.0005		0.6-3.0
g - Chlordane	0.0043	0.00081	0.000081	TBD	0.0005		0.6-3.0
a - Chlordane	0.0043	0.00081	0.000081	TBD	0.0005		0.6-3.0
Endosulfan I	0.051	0.051	8.9	TBD	0.0005		0.6-3.0
4,4'-DDE	0.2	0.00022	0.000022	TBD	0.0005		0.6-3.0
Dieldrin	0.0042	0.000054	0.0000054	TBD	0.0005		0.6-3.0
Endrin	0.036	0.036	0.006	TBD	0.0005		0.6-3.0
Endosulfan II	0.051	0.051	0.051	TBD	0.0005		0.6-3.0
4,4'-DDD	0.280	0.00031	0.000031	TBD	0.0005		0.6-3.0
Endrin aldehyde	NE	0.3	0.03	TBD	0.0005		0.6-3.0
4,4'-DDT	0.001	0.00022	0.000022	TBD	0.0005		0.6-3.0
Endosulfan sulfate	NE	89	8.9	TBD	0.0005		0.6-3.0
Endrin ketone	NE	NE	NE	TBD	0.0005		0.6-3.0
Methoxychlor	0.019	0.019	0.019	TBD	0.0005		0.6-3.0
Hexachlorobenzene	0.042	0.00029	0.000029	TBD	0.0005		0.6-3.0
Toxaphene	0.0002	0.0002	0.000028	TBD	0.025		0.6-3.0
Hexachlorobutadiene	0.86	0.86	0.86	TBD	0.001		0.6-3.0
oxy chlordane	0.19	0.19	0.19	TBD	0.0005		0.6-3.0
cis - nonachlor	0.19	0.19	0.19	TBD	0.0005		0.6-3.0
trans - nonachlor	0.19	0.19	0.19	TBD	0.0005		0.6-3.0
2,4'-DDD	0.28	0.28	0.28	TBD	0.0005		0.6-3.0
2,4'-DDE	0.2	0.2	0.2	TBD	0.0005		0.6-3.0
2,4'-DDT	0.2	0.2	0.2	TBD	0.0005		0.6-3.0
<b>Semivolatile Organic Compounds, µg/L (ppb)</b>							
<b>Halogenated Compounds</b>							
1,2-Dichlorobenzene	14	14	14	0.014	0.2		
1,3-Dichlorobenzene	5.5	5.5	5.5	0.011	0.2		

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
				Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
1,4-Dichlorobenzene	0.5	0.5	0.5	0.014	0.2		
1,2,4-Trichlorobenzene	50	50	7	0.016	0.2		
Hexachlorobenzene	0.042	0.00029	0.000029	0.014	0.2		
2-Chloronaphthalene	490	490	160	0.015	0.2		
Hexachloroethane	4.8	303.0	0.33	0.018	0.2		
Hexachlorobutadiene	0.86	0.86	0.86	0.019	0.2		
Hexachlorocyclopentadiene	5.2	5.2	5.2	0.041	1		
2,2'-oxybis(1-chloropropane)	NE	NE	NE	0.017	0.2		
Bis-(2-chloroethoxy) methane	11000	11000	11000	0.012	0.2		
Bis-(2-chloroethyl) ether	0.0098	0.0098	0.0098	0.014	0.2		
4-Chlorophenyl-phenyl ether	NE	NE	NE	0.0084	0.2		
4-bromophenyl-phenyl ether	1.5	1.5	1.5	0.018	0.2		
3,3'-Dichlorbenzidine	0.15	0.028	0.0028	0.43	2		
4-Chloroaniline	50	50	50	0.017	0.2		
<b>Organonitrogen Compounds</b>							
Nitrobenzene	3.4	3.4	3.4	0.0074	0.2		
Aniline	12	12	12	TBD	1		
2-Nitroaniline	1.0	1.0	1.0	0.015	0.2		
3-Nitroaniline	NE	NE	NE	0.23	1		
4-Nitroaniline	NE	NE	NE	0.16	1		
N-Nitrosodimethylamine	0.0013	0.0013	0.0013	0.00026	0.002		
N-Nitroso-di-n-propylamine	0.0096	0.0096	0.0096	0.032	0.2		
N-Nitrosodiphenylamine	14	6	0.6	0.028	0.2		
2,4-Dinitrotoluene	73	3.4	0.34	0.019	0.2		
2,6-Dinitrotoluene	36	36	36	0.0088	0.2		
Carbazole	3.4	3.4	3.4	0.013	0.2		
<b>Oxygen-Containing Compounds</b>							
Benzoic Acid	42	42	42	1.71	5		
Benzyl Alcohol	8.6	8.6	8.6	0.97	5		

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
				MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
Dibenzofuran	3.7	3.7	3.7	0.013	0.2		
Isophorone	71	71	71	0.0084	0.2		
<b>Phenols and Substituted Phenols</b>							
Phenol	22000	22000	22000	0.020	0.5		
2-Methylphenol	13	13	13	0.059	0.5		
4-Methylphenol	180	180	180	0.051	0.5		
2,4-Dimethylphenol	730	730	85	0.32	2		
2-Chlorophenol	30	30	15	0.015	0.5		
2,4-Dichlorophenol	110	110	29	0.024	0.5		
2,4,5-Trichlorophenol	63	63	63	0.025	0.5		
2,4,6-trichlorophenol	3.6	2.4	0.24	0.037	0.5		
2,3,4,6-Tetrachlorophenol	1100	1100	1100	TBD	TBD		
Pentachlorophenol	0.56	0.56	0.3	0.028	1		
4-Chloro-3-methylphenol	NE	NE	NE	0.029	0.5		
2-Nitrophenol	NE	NE	NE	0.014	0.5		
4-Nitrophenol	150	150	150	0.54	2		
2,4-Dinitrophenol	73	73	73	0.53	4		
4,6-Dinitro-2-methylphenol	NE	280	28	0.013	2		
<b>Phthalate Esters</b>							
Dimethylphthalate	3	3	3	0.013	0.2		ng/L (ppt)
Diethylphthalate	3	3	3	0.026	0.2		0.3 - 3.0
Di-n-butylphthalate	1	1	1	0.026	0.2		0.3 - 3.0
Butylbenzylphthalate	3	3	3	0.025	0.2		0.3 - 3.0
Di-n-octylphthalate	3	3	3	0.032	0.2		0.3 - 3.0
bis(2-Ethylhexyl)phthalate	0.12	0.12	0.12	0.27	2		0.3 - 3.0
<b>Polycyclic Aromatic Hydrocarbons</b>							
Naphthalene	6.2	6.2	6.2	0.014	0.02		ng/L (ppt)
2-Methylnaphthalene	NE	NE	NE	0.012	0.02		0.1-0.6
Acenaphthylene	NE	NE	NE	0.0089	0.02		0.1-0.6

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
				Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
Acenaphthene	23	23	23	0.0097	0.02		
Fluorene	3.9	3.9	3.9	0.011	0.02		0.1-0.6
Phenanthrene	6.3	6.3	6.3	0.013	0.02		0.1-0.6
Anthracene	0.09	0.09	0.09	0.010	0.02		0.1-0.6
Fluoranthene	6.2	6.2	6.2	0.013	0.02		0.1-0.6
Pyrene	180	180	180	0.012	0.02		0.1-0.6
Benzo(a)anthracene	0.027	0.018	0.0018	0.013	0.02		0.1-0.6
Chrysene	9.2	0.018	0.0018	0.012	0.02		0.1-0.6
Benzo(b)fluoranthene	0.092	0.018	0.0018	0.0098	0.02		0.1-0.6
Benzo(k)fluoranthene	0.92	0.018	0.0018	0.011	0.02		0.1-0.6
Benzo(a)pyrene	0.0092	0.0092	0.0018	0.0087	0.02		0.1-0.6
Indeno(1,2,3-cd)pyrene	0.092	0.018	0.0018	0.0087	0.02		0.1-0.6
Dibenz(a,h)anthracene	0.0092	0.0092	0.0018	0.0079	0.02		0.1-0.6
Benzo(g,h,i)perylene	NE	NE	NE	0.0090	0.02		0.1-0.6
<b>Chlorinated Dioxins and Furans, µg/L (ppb)</b>							<b>pg/L (ppq)</b>
2,3,7,8,-TCDD (Toxicity Equivalence Quotient)	4.5E-07	5.1E-09					
2,3,7,8,-TCDD							0.003 - 0.01
2,3,7,8-TCDF							0.003 - 0.01
1,2,3,7,8-PeCDD							0.003 - 0.01
1,2,3,7,8-PeCDF							0.003 - 0.01
2,3,4,7,8-PeCDF							0.003 - 0.01
1,2,3,4,7,8-HxCDD							0.003 - 0.01
1,2,3,6,7,8-HxCDD							0.003 - 0.01
1,2,3,7,8,9-HxCDD							0.003 - 0.01
1,2,3,4,7,8-HxCDF							0.003 - 0.01
1,2,3,6,7,8-HxCDF							0.003 - 0.01
1,2,3,7,8,9-HxCDF							0.003 - 0.01
2,3,4,6,7,8-HxCDF							0.003 - 0.01



Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

Analytes	Analytical Concentration Goals			Laboratory MDLs and MRLs			
	Level 1 ACG <sup>2</sup>	Level 2 ACG <sup>3</sup>	Level 3 ACG <sup>4</sup>	Peristaltic Pump Samples		XAD-2 Samples <sup>1</sup>	
				MDL (CAS, NEA)	MRL (CAS, NEA)	MRL (NEA)	MDL (Axys)
1,2,3,4,6,7,8-HpCDD							0.003 - 0.01
1,2,3,4,6,7,8-HpCDF							0.003 - 0.01
1,2,3,4,7,8,9-HpCDF							0.003 - 0.01
OCDD							0.003 - 0.01
OCDF							0.003 - 0.01
Total tetrachlorinated dioxins							0.003 - 0.01
Total pentachlorinated dioxins							
Total hexachlorinated dioxins							
Total heptachlorinated dioxins							
Total tetrachlorinated furans							
Total pentachlorinated furans							
Total hexachlorinated furans							
Total heptachlorinated furans							
PCB Congeners							pg/L (ppq)
All 209 PCB congener target analytes							0.03-3

Table A6-2 Analytes, Analytical Concentration Goals, and Method Reporting Limits for Surface Water Samples.

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Note

- <sup>1</sup> MDLs are provided for XAD filtrate and particulate samples. The particulate samples will be reported on a pg/L basis calculated from the volume of water pumped through the filter during sample collection.
- <sup>2</sup> Level 1 ACGs are the lowest of the EPA Region 9 PRGs for Tap Water (EPA 2002b), NRWQC freshwater aquatic life criteria (EPA 2002c), or ORNL values (Suter and Tsao 1996).
- <sup>3</sup> Level 2 ACGs are the lowest of the EPA Region 9 PRGs for Tap Water (EPA 2002b), NRWQC freshwater aquatic life criteria and human health criteria (EPA 2002c), ORNL values (Suter and Tsao 1996), and the fish consumption criteria from the Revised Human Health Water Quality Criteria (EPA 2003).
- <sup>4</sup> Level 3 ACGs are the lowest of the EPA Region 9 PRGs for Tap Water (EPA 2002b), NRWQC freshwater aquatic life criteria and human health criteria (EPA 2002c), ORNL values (Suter and Tsao 1996), the subsistence fish consumption criteria from the Revised Human Health Water Quality Criteria (EPA 2003), and site-specific subsistence fish consumption criteria.
- <sup>5</sup> Required for natural attenuation evaluation (Anchor Environmental 2004)
- <sup>6</sup> Parameters for calculating freshwater dissolved metals criteria that are hardness-dependent are from NRWQC (EPA 2002c). Hardness dependent criteria based on average hardness of 25 mg/L (CaCO<sub>3</sub>) (USGS database from 1974 to 1990).
- <sup>7</sup> Based on Notice of Availability of Final Aquatic Life Criteria Document for Tributyltin (69 Fed. Reg. 2, 342).

MRL = minimum reporting limit

NA = not applicable

NE = not established

NRWQC = National Recommended Water Quality Criteria

ORNL = Oak Ridge National Laboratory

PRG = preliminary remediation goals

TBD = to be determined

Table A7-1. Laboratory Control Limits for Surrogate Samples.

Analysis	Percent Recovery
<b>Butyltins</b>	
Tri-n-propyltin	19-146
<b>Chlorinated Herbicides</b>	
2,4-Dichlorophenylacetic acid	15-126
<b>Organochlorine Pesticides</b>	
Tetra-chloro-meta-xylene	60-140
Decachlorobiphenyl	60-140
<b>PCB Aroclors</b>	
Tetra-chloro-meta-xylene	30-150
Decachlorobiphenyl	30-150
<b>Semivolatile Organic Compounds</b>	
2-Fluorophenol	33-109
Phenol-d6	41-118
2,4,6-Tribromophenol	34-130
Nitrobenzene-d5	39-120
2-Fluorobiphenyl	36-107
Terphenyl-d14	38-148
<b>Semivolatile Organic Compounds (SIM)</b>	
Fluoranthene-d10	18-137
Fluorene-d10	37-107
Terphenyl-d14	18-153
<b>XAD-2 Resin (Analysis will be conducted on filtrate and solid material collected on filter)</b>	
<b>PCB Aroclors</b>	
Tetra-chloro-meta-xylene	30-150
Decachlorobiphenyl	30-150
<b>Organochlorine Pesticides</b>	
<sup>13</sup> C <sub>6</sub> -delta-HCH, <sup>13</sup> C <sub>12</sub> -Dieldrin, <sup>13</sup> C <sub>12</sub> -Endrin, <sup>13</sup> C <sub>10</sub> -Heptachlor epoxide, <sup>13</sup> C <sub>12</sub> -Methoxychlor, <sup>13</sup> C <sub>9</sub> -alpha-Endosulphan, <sup>13</sup> C <sub>9</sub> -beta-Endosulphan, <sup>13</sup> C <sub>6</sub> -beta-HCH, <sup>13</sup> C <sub>6</sub> -delta-HCH, <sup>13</sup> C <sub>6</sub> -gamma-HCH, <sup>13</sup> C <sub>10</sub> -Heptachlor, <sup>13</sup> C <sub>12</sub> -Aldrin, <sup>13</sup> C <sub>10</sub> -trans-Nonachlor, <sup>13</sup> C <sub>10</sub> -cis-nonachlor, <sup>13</sup> C <sub>10</sub> -Mirex	30-150
<sup>13</sup> C <sup>12</sup> -c,p'-DDE, <sup>13</sup> C <sub>12</sub> -p,p'-DDE, <sup>13</sup> C <sub>12</sub> -c,p'-DDT, <sup>13</sup> C <sub>12</sub> -p,p'-DDT	40-150
<sup>13</sup> C <sub>6</sub> -Pentachlorobenzene, <sup>13</sup> C <sub>6</sub> -Hexachlorobenzene	20-150
<sup>13</sup> C <sub>6-1,2,3</sub> -trichlorobenzene, <sup>13</sup> C <sub>6</sub> -1,2,3,4-tetrachlorobenzene	20-130
<sup>13</sup> C <sub>10</sub> -Oxhchlorodane, <sup>13</sup> C <sub>10</sub> -trans-chlordane	30-200
<sup>13</sup> C <sub>6</sub> -Chlorobenzene	10-130
<sup>13</sup> C <sub>6</sub> -C1,4-dichlorobenzene	15-130

Table A7-1. Laboratory Control Limits for Surrogate Samples.

Analysis	Percent Recovery
<b>SVOCs (phthalate esters and PAHs)</b>	
Phthalate esters all surrogates	40-140
<b>PAHs</b>	
d10-phenanthrene, d1--fouoranthene, d12-benz(a)anthracene, d12-chrysene, d12-benzo(bk)fluoranthene, d12-benao(a)pyrene, d12-perylene	30-130
d14-dibenz(ah)anthracene, d12-indeno91,2,3-cd)pyrene, d12- benao(ghi)perylene, d10-2methylnaphthalene, d12-2,6dimethylnaphthalene	30-120
d8-naphthalene	15-130
d10-acenaphthylene, d10-2methylnaphthalene, d12-2,6dimethylnaphthalene	20-130
<b>Dioxins and Furans</b>	
Suite of 13C-labeled surrogates	17-130 <sup>1</sup>

Note:

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

<sup>1</sup> Laboratory control limits as per EPA1613B apply to individual PCDD/F labeled surrogates.

Table A7-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples.

Analysis	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
<b>Conventional Analyses</b>				
Total suspended solids	NA	NA	LD	20
Total dissolved solids	NA	NA	LD	20
Total organic carbon	76-121	92-106 <sup>1</sup>	LD	20
Dissolved organic carbon	76-121	92-106 <sup>1</sup>	LD	20
Perchlorate	80-120	85-115	LD	20
<b>Metals</b>				
Aluminum, antimony, cadmium, total chromium, copper, lead, nickel, selenium, silver, zinc	70-130	85-115 <sup>2</sup>	LD	20
Arsenic	70-130	85-115 <sup>2</sup>	LD	20
Mercury	85-115	84-114	LD	20
Hexavalent chromium	85-115	85-115 <sup>1</sup>	LD	25
Hardness (Ca, Mg)	70-130	85-115 <sup>2</sup>	LD	20
<b>Butyltins</b>				
Monobutyltin	10-151	13-167	MSD	30
Dibutyltin	10-151	10-128	MSD	30
Tributyltin	14-143	11-145	MSD	30
Tetrabutyltin	Oct-93	10-112	MSD	30
<b>Chlorinated Herbicides</b>				
Dalapon	10-172	13-162	MSD	30
Dicamba	27-149	29-140	MSD	30
MCPA	10-157	10-156	MSD	30
Dichlorprop	10-172	10-159	MSD	30
2,4-D	37-130	24-139	MSD	30
2,4,5-TP (Silvex)	10-148	17-139	MSD	30
2,4,5-T	10-121	14-118	MSD	30
2,4-DB	10-170	10-171	MSD	30
Dinoseb	10-170	10-155	MSD	30
MCPP	10-151	12-143	MSD	30
<b>Organochlorine Pesticides</b>				
All target analytes	50-150	60-140	MSD	20
<b>PCB Aroclors</b>				
All target analytes	50-150	60-140	MSD	20
<b>Semivolatile Organic Compounds</b>				
Phenol	40-111	45-119	MSD	30
2-Chlorophenol	44-109	43-120	MSD	30
Bis(2-chloroethyl)ether	42-111	41-124	MSD	30
1,3-Dichlorobenzene	13-75	16-86	MSD	30
1,4-Dichlorobenzene	13-81	17-86	MSD	30
1,2-Dichlorobenzene	16-89	21-93	MSD	30
2-Methylphenol	37-110	30-120	MSD	30
Bis(2-chloroisopropyl)ether	32-112	35-120	MSD	30
4-Methylphenol	32-115	27-123	MSD	30
Hexachloroethane	10-89	10-78	MSD	30

Table A7-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples.

Analysis	Matrix Spike	Laboratory Control Sample	Type of Duplicate	Control Limit
	Recovery (percent)	Recovery (percent)		Relative Percent Difference
N-Nitroso-di-n-propylamine	40-130	40-134	MSD	30
Nitrobenzene	23-149	28-126	MSD	30
Isophorone	42-133	46-135	MSD	30
2,4-Dimethylphenol	10-125	10-105	MSD	30
2-Nitrophenol	44-117	44-124	MSD	30
Bis(2-chloroethoxy)methane	40-115	41-120	MSD	30
2,4-Dichlorophenol	44-114	43-120	MSD	30
1,2,4-Trichlorobenzene	16-89	21-90	MSD	30
Hexachlorobutadiene	10-81	10-80	MSD	30
Naphthalene	10-125	27-116	MSD	30
4-Chloroaniline	10-85	10-110	MSD	30
4-Chloro-3-methylphenol	41-120	33-126	MSD	30
2-Methylnaphthalene	10-121	22-106	MSD	30
2,4,6-Trichlorophenol	46-115	48-116	MSD	30
2,4,5-Trichlorophenol	45-116	46-120	MSD	30
2-Chloronaphthalene	26-120	33-121	MSD	30
2-Nitroaniline	10-148	41-127	MSD	30
Dimethyl phthalate	52-117	48-125	MSD	30
Acenaphthylene	18-135	33-131	MSD	30
2,6-Dinitrotoluene	45-129	47-130	MSD	30
3-Nitroaniline	10-125	41-128	MSD	30
Acenaphthene	10-139	31-122	MSD	30
4-Nitrophenol	31-140	39-126	MSD	30
Dibenzofuran	10-138	31-119	MSD	30
Diethylphthalate	44-136	45-140	MSD	30
2,4-Dinitrophenol	10-178	14-131	MSD	30
2,4-Dinitrotoluene	51-129	51-134	MSD	30
4-Chlorophenyl phenyl ether	37-107	40-119	MSD	30
Fluorene	10-147	33-120	MSD	30
N-nitrosodiphenylamine	36-123	43-131	MSD	30
4-Nitroaniline	10-130	44-127	MSD	30
4,6-Dinitro-2-methylphenol	30-127	31-123	MSD	30
4-Bromophenyl phenyl ether	36-122	40-127	MSD	30
Hexachlorobenzene	40-120	42-124	MSD	30
Pentachlorophenol	22-143	23-125	MSD	30
Phenanthrene	15-146	35-127	MSD	30
Anthracene	15-137	34-126	MSD	30
Carbazole	44-122	51-125	MSD	30
Di-n-butyl phthalate	48-124	47-134	MSD	30
Fluoranthene	35-125	36-132	MSD	30
Pyrene	15-146	38-129	MSD	30
Butyl benzyl phthalate	50-121	50-128	MSD	30
Bis(2-ethylhexyl) phthalate	22-155	47-140	MSD	30
3,3'-Dichlorobenzidine	70-130	24-106	MSD	30
Benzo(A)anthracene	23-134	39-128	MSD	30
Chrysene	34-122	40-128	MSD	30
Di-n-octyl phthalate	45-124	47-134	MSD	30
Benzo(B)fluoranthene	31-125	36-135	MSD	30
Benzo(K)fluoranthene	36-121	38-133	MSD	30
Benzo(A)pyrene	29-118	35-129	MSD	30
Dibenzo(A,H)Anthracene	30-133	38-135	MSD	30
Indeno(1,2,3-C,D) Pyrene	31-126	37-133	MSD	30
Benzo(G,H,I) perylene	20-141	39-133	MSD	30
Benzoic acid	10-133	10-86	MSD	30
Benzyl alcohol	46-113	37-128	MSD	30

Table A7-2. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples.

Analysis	Matrix Spike Recovery (percent)	Laboratory Control Sample Recovery (percent)	Type of Duplicate	Control Limit Relative Percent Difference
<b>XAD-2 Resin (Analysis will be conducted on filtrate and solid material collected on filter)</b>				
<b>PCB Aroclors</b>	NA	50-150	NA <sup>5</sup>	NA
<b>Organochlorine Pesticides</b>				
Target compounds	NA	60-130/70-130 <sup>3</sup>	NA <sup>5</sup>	NA
<b>SVOCs</b>				
Phthalate esters (all target compounds)	NA	70-130	NA <sup>5</sup>	NA
PAHs	NA	70-130 <sup>4</sup>	NA <sup>5</sup>	NA
<b>Dioxins and Furans</b>	NA	NA	NA <sup>5</sup>	NA
<b>PCB congeners</b>	NA	NA	NA <sup>5</sup>	NA

**Notes**

LD - laboratory duplicate

MSD - matrix spike duplicate

LCSD - laboratory control sample duplicate

NA - Not applicable

Control limits are updated periodically by the laboratories and may differ slightly from the control limits shown in this table. During data validation, low recoveries will be evaluated for analytes with wide laboratory control limits to evaluate potential bias of the data. Data will be qualified if a bias is identified.

<sup>1</sup> In-house limits

<sup>2</sup> Method specified control limits

<sup>3</sup> Laboratory control limits for delta-HCH, heptachlor epoxide, dieldrin, endrin, endrin ketone, methoxychlor, dichlorobenzenes, trichlorobenzenes, and tetrachlorobenzenes is 60-130. Laboratory control limits for all other target compounds is 70-130.

<sup>4</sup> Control limits for Acenaphthylene are 70-140

<sup>5</sup> Field duplicates will be used to assess overall precision. Laboratory duplicates cannot be used because of sample limitations

Table B2-1. Sample Containers and Preservation Requirements for Surface Water Samples.

	Container		Additional for Lab QC <sup>1</sup>	Preservation	Holding Time	Laboratory Sample Size
	Type	Size				
<b>Sample Collected by Peristaltic Pump<sup>2</sup></b>						
Total Suspended Solids	HDPE	1 liter	2 liters	4±2°C	7 days	1 liter
Total Dissolved Solids	HDPE	500 mL	500 mL	4±2°C	7 days	250 ml
Total Organic Carbon	HDPE	250 mL	none	H <sub>2</sub> S0 <sub>4</sub> to pH <2; 4±2°C	28 days	50 ml
Dissolved Organic Carbon	HDPE	250 mL	none	H <sub>2</sub> S0 <sub>4</sub> to pH <2; 4±2°C	28 days	50 ml
Unfiltered metals	HDPE	1 liter	1 liter	5 ml of 1:1& HNO <sub>3</sub> ; 4±2°C	6 months/60 days <sup>3</sup>	300 ml
Filtered metals and hardness	HDPE	1 liter	1 liter	5 ml of 1:1& HNO <sub>3</sub> ; 4±2°C	6 months/60 days <sup>3</sup>	300 ml
Hexavalent chromium	HDPE	500 mL	none	4±2°C	24 hours	100 ml
Butyltin compounds	PC	1 liter	2 liters	4±2°C	7 days	1 liter
Perchlorate	HDPE	250 mL	none	4±2°C	28 days	10 mL
Chlorinated herbicides	AG	1 liter	2 liters	4±2°C	7 /40 days <sup>4</sup>	1 liter
Chlorinated pesticides	AG	1 liter	2 liters	4±2°C	7 /40 days <sup>4</sup>	1 liter
Polychlorinated biphenyls	AG	4 liters	8 liters	4±2°C	7 /40 days <sup>4</sup>	4 liters
Semivolatile organic compounds	AG	1 liter	2 liters	4±2°C	7 /40 days <sup>4</sup>	1 liter
Chlorinated dioxins and furans	AG	1 liter	2 liters	4±2°C	30/45 days <sup>5</sup>	1 liter
<b>XAD-2 Resin</b>	XAD	250 g	1 column	0-4°C		
Chlorinated pesticides					NE <sup>6</sup>	1/6 of extract
Polychlorinated biphenyls					NE <sup>6</sup>	1/6 of extract
Phthalate esters					NE <sup>6</sup>	1/6 of extract
Polycyclic aromatic hydrocarbons					NE <sup>6</sup>	1/6 of extract
Chlorinated dioxins and furans					NE <sup>6</sup>	2/6 of extract
<b>Filtered Particulate for XAD-2 Resin</b>	Filter cartridge will be placed		NA	0-4°C		
Chlorinated pesticides and PCB congeners	in 16-oz. amber glass sample jar				NE <sup>6</sup>	1/6 of extract
Polychlorinated biphenyl Aroclors®					NE <sup>6</sup>	1/6 of extract
Phthalate esters					NE <sup>6</sup>	1/6 of extract
Polycyclic aromatic hydrocarbons					NE <sup>6</sup>	1/6 of extract
Chlorinated dioxins and furans					NE <sup>6</sup>	2/6 of extract



Table B2-1. Sample Containers and Preservation Requirements for Surface Water Samples.

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HDPE = High density polyethylene bottle

PC = Polycarbonate bottle

AG = Amber glass bottle with teflon-lined lid

XAD = XAD-2 resin in stainless steel column

NE = Not established

<sup>1</sup>Sample container sizes may be modified to meet laboratory requirements.

<sup>2</sup>Extra sample volume will be collected at a frequency of 5% of samples to accommodate requirements for laboratory QC samples .

<sup>3</sup>The holding time for mercury is 60 days, based on CRITFC study (EPA 2002a) and EPA Method 1631 revision D (EPA 2001a).  
The holding time for the remaining metals is 6 months.

<sup>4</sup>The holding time is 7 days from collection to extraction and 40 days from extraction to analysis.

<sup>5</sup>The holding time is 30 days from collection to extraction and 45 days from extraction to analysis.

<sup>6</sup> A technical holding time has not been established for XAD extracts and filters. The contractual holding time is 30 days from collection to extraction and 30 days from extraction to analysis.

Table B4-3. Summary of Surface Water and Field Quality Control Samples for Each Surface Water Sampling Event.

Parameter	Samples	Sample Splits <sup>1,2</sup>	Field Replicates <sup>1</sup>	Field Rinsate Blanks <sup>3,4</sup>	Decon Blank <sup>5</sup>	Total Number of Field Samples
<b><u>Early Fall Sampling Event</u></b>						
<b><i>Peristaltic Pump</i></b>						
Metals (unfiltered)	23	2	2	2	1	30
Metals (filtered)	23	2	2	2	1	30
PCB Aroclors	16	1	1	2	1	21
Chlorinated Herbicides	23	2	2	2	1	30
Organochlorine Pesticides	16	1	1	2	1	21
SVOCs	23	2	1	2	1	29
Butyltin compounds	23	2	2	2	1	30
Perchlorate	1	1	1	1	1	5
TSS	23	2	2	0	0	27
TDS	23	2	2	0	0	27
DOC	23	2	2	2	0	29
TOC	23	2	2	2	0	29
Hardness	23	2	2	2	0	29
<b><i>XAD-2 Resin (Analysis will be conducted on filtrate and solid material collected on filter)</i></b>						
PCB Aroclors <sup>5</sup>	7	0	1	1	0	9
Organochlorine Pesticides	7	0	1	1	0	9
SVOCs (phthalate esters and PAHs)	7	0	1	1	0	9
Dioxins/Furans	5	0	1	1	0	7
<b><u>Late Fall Sampling Event</u></b>						
<b><i>Peristaltic Pump</i></b>						
Metals (unfiltered)	23	2	2	2	1	30
Metals (filtered)	23	2	2	2	1	30
PCB Aroclors	16	1	1	2	1	21
Chlorinated Herbicides	23	2	2	2	1	30
Organochlorine Pesticides	16	1	1	2	1	21
SVOCs	23	2	1	2	1	29
Butyltin compounds	23	2	2	2	1	30
Perchlorate	1	1	1	1	1	5
TSS	23	2	2	0	0	27
TDS	23	2	2	0	0	27
DOC	23	2	2	2	0	29
TOC	23	2	2	2	0	29
Hardness	23	2	2	2	0	29
<b><i>XAD-2 Resin (Analysis will be conducted on filtrate and solid material collected on filter)</i></b>						
PCB Aroclors <sup>5</sup>	7	0	1	1	0	9
Organochlorine Pesticides	7	0	1	1	0	9
SVOCs (phthalate esters and PAHs)	7	0	1	1	0	9
Dioxins/Furans	5	0	1	1	0	7

Table B4-3. Summary of Surface Water and Field Quality Control Samples for Each Surface Water Sampling Event.

Parameter	Samples	Sample Splits <sup>1,2</sup>	Field Replicates <sup>1</sup>	Field Rinsate Blanks <sup>3,4</sup>	Decon Blank <sup>5</sup>	Total Number of Field Samples
<b><u>Late Winter High-Flow Sampling Event</u></b>						
<b><i>Peristaltic Pump</i></b>						
Metals (unfiltered)	23	2	2	2	1	30
Metals (filtered)	23	2	2	2	1	30
PCB Aroclors	20	1	1	2	1	25
Chlorinated Herbicides	23	2	1	2	1	29
Organochlorine Pesticides	20	1	1	2	1	25
SVOCs	23	2	1	2	1	29
Butyltin compounds	23	2	1	2	1	29
Perchlorate	1	1	1	1	1	5
TSS	23	2	2	0	0	27
TDS	23	2	2	0	0	27
DOC	23	2	2	2	0	29
TOC	23	2	2	2	0	29
Hardness	23	2	2	2	0	29
<b><i>XAD-2 Resin (Analysis will be conducted on filtrate and solid material collected on filter)</i></b>						
PCB Aroclors <sup>6</sup>	3	0	1	1	0	5
Organochlorine Pesticides	3	0	1	1	0	5
SVOCs (phthalate esters and PAHs)	3	0	1	1	0	5
Dioxins/Furans	3	0	1	1	0	5

Note:

<sup>1</sup> The collection frequency for field replicates and splits is 5% of natural samples.

<sup>2</sup> At EPA's discretion, the field splits may be analyzed by EPA rather than LWG.

<sup>3</sup> A field rinsate blank will be collected for the peristaltic pump near the beginning and end of each sampling event.

<sup>4</sup> An uninstalled XAD-2 resin column and a filter will travel with the sampling team. The column and filter will be archived at the laboratory and analyzed if irregularities are noted in the data for the surface water samples

<sup>5</sup> Prior to the start of sample collection activities, a decon blank will be generated by the laboratory that conducts decontamination of the peristaltic pump sampling equipment.

<sup>6</sup> An aliquot of the XAD extracts will be archived for potential PCB congener analysis.

# **APPENDIX A**

## **AXYS'S QUALITY ASSURANCE MANUAL**

(The Axy's QA Manual will be supplied to the EPA QA Manager under separate cover.)

# **APPENDIX B**

## **DECONTAMINATION OF FIELD SAMPLING EQUIPMENT FOR SURFACE WATER SAMPLING SOP**

## DECONTAMINATION OF FIELD SAMPLING EQUIPMENT FOR SURFACE WATER SAMPLING

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The purpose of this standard operating procedure (SOP) is to define and standardize the methods for decontamination of field sampling equipment for the collection of surface water samples using a peristaltic pump and Teflon™ tubing to ensure sample integrity and minimize contamination during sample handling. Details on surface water sampling techniques and equipment setup are described in Appendix C “Surface Water Sampling SOP” of the Portland Harbor RI/FS Round 2 Field Sampling Plan, Surface Water Sampling (Integral, 2004).

This SOP utilizes and augments the procedures outlined in the *Interagency Field Manual for the Collection of Water-Quality Data* (USGS 2000) and U.S. Environmental Protection Agency (EPA) Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA 1996). Clean sampling techniques designed for trace metals will be used for the collection of filtered and unfiltered water samples. Samples will be analyzed for organic compounds, metals, and conventionals for the surface water sampling events.

## SUMMARY OF METHOD

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Preparation of the Teflon™ sample tubing will be completed in the laboratory 3-4 weeks before the start of a sampling event. All sample tubing, filters and containers will be decontaminated, pre-assembled and packed for the field in double bagged polyethylene bags.

Decontamination of tubing, filters, stir bars, and containers consists of soaking the sampling equipment in cleaning solutions for a pre-determined amount of time and then rinsing it with deionized water. A peristaltic pump is used to fill the Teflon™ sample tubing with cleaning solutions and to flush with deionized water between cleanings. In similar fashion, sampling filters are also attached in series with C-flex™ tubing, filled with cleaning solutions and flushed with deionized water between cleanings. The Teflon™ tubing ends and in-line filters are joined together with C-flex™ tubing for transport to the field.

## PROCEDURES

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The procedure that will be used for each type of sampling equipment is provided below. Selected equipment (e.g., tubing) will be used for collection of all target parameters. However, selected equipment (containers, filter units) will be processed separately for metals and organics.

### **Teflon™ and C-Flex™ Pump Tubing and Teflon™ Coated Stir Bars for Intake Water**

The Teflon™ and C-Flex™ tubing used for the intake of site water and Teflon coated stir bars used in the containers will be cleaned for all target parameters. Cleaning procedures are as follows:

- Completely fill sample tubing with reagent grade methanol and let it soak for 24 hours. Soak stir bars in reagent grade methanol for 24 hours.
- Drain methanol solution from the tubing and stir bars and flush with approximately 10 liters (L) of deionized (DI) water.
- Completely fill sample tubing with dilute reagent grade nitric acid (HNO<sub>3</sub>) and let soak for 24 hours. Soak stir bars in reagent grade HNO<sub>3</sub> for 24 hours.
- Drain acid and flush tubing and stir bars with DI water. Fill tubing with DI water, and let set for 24 hours. Soak stir bars in DI water for 24 hours. Drain, rinse with DI water, and drain.
- Double bag clean tubing and stir bars, label outside bag identifying cleaning process used, and store in double-bagged polyethylene bags until assembly.

### **Teflon™ and C-Flex™ Pump Tubing From Filtration Units to Sample Jars**

The Teflon™ and C-Flex™ tubing used from the filtration units to the sample jars will be cleaned for metals or for organic and conventional parameters, as described below:

#### **Metals, Butyltin, and Conventional Analysis**

- Completely fill sample tubing with dilute reagent grade HNO<sub>3</sub> and let soak for 24 hours.
- Drain acid and flush tubing with DI water, fill with DI water, and let set for 24 hours. Drain, rinse with DI water, and drain.
- Double bag clean tubing, label outside bag identifying cleaning process used, and store in double-bagged polyethylene bags until assembly.

#### **Organic Analysis**

- Completely fill sample tubing with reagent grade methanol and let it soak for 24 hours.
- Drain methanol solution from the tubing and flush with approximately 10 L of DI water.
- Double bag clean tubing, label outside bag identifying cleaning process used, and store in double-bagged polyethylene bags until assembly.

## **Filter Cartridges**

Filter cartridges (0.45 µm and 10 µm) will be used for sample collection. The 0.45 µm cartridges will be used for the collection of dissolved trace metal samples and DOC in water. The filter size for DOC will be selected in consultation with EPA. The DOC filter cartridge will be cleaned according to the same procedure used for the 0.45 µm and 10 µm cartridges. The 10 µm cartridges will be used for all samples. Normally, one filter cartridge is used per sample site, although elevated suspended solids concentrations may require using two or more cartridges per site. The following procedures are used for cleaning the 10 µm and 0.45 µm filter cartridges for metals, butyltin species, and conventionals, and the selected cartridge for DOC:

- Completely fill filter cartridge with dilute reagent grade HNO<sub>3</sub> and let soak for 24 hours.
- Drain acid and flush filter cartridge with DI water, fill with DI water, and let set for 24 hours. Drain, rinse with DI water, and drain.
- Double bag clean cartridges, label outside bag with cleaning process used, and store in double-bagged polyethylene bags until assembly.

The following procedures are used for cleaning the 10 µm filter cartridges for organics:

- Completely fill filter cartridge with reagent grade methanol and let it soak for 24 hours.
- Drain methanol solution from the filter cartridge and flush with approximately 10 L of DI water.
- Double bag clean cartridges, label outside bag with cleaning process used, and store in double-bagged polyethylene bags until assembly .

## **Glass and Polycarbonate 20 Liter Containers:**

The glass and polycarbonate containers will be used to composite the sample prior to distribution to the sample containers. The cleaning procedure for metals and for organic and conventional parameters is described below:

### **Metals, Butyltin, and Conventional Analysis**

- Completely fill polycarbonate container with dilute reagent grade HNO<sub>3</sub> and let soak for 24 hours.



- Drain acid and flush container with DI water, fill with DI water, and let set for 24 hours. Drain, rinse with DI water, and drain.
- Assemble components of filtration unit and store in double-bagged polyethylene bags until used in the field .

### **Organic Analysis**

- Completely fill glass container with reagent grade methanol and let it soak for 24 hours.
- Drain methanol solution from the tubing and flush with DI water.
- Assemble components of filtration unit and store in double-bagged polyethylene bags until used in the field .

## **ASSEMBLY**

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After all the components have been cleaned, the peristaltic pump collection and filtration units will be assembled by the laboratory performing the sample equipment decontamination. The assembled units will be double-bagged in polyethylene bags, labeled for identification on the outside bag, and sent to the field laboratory.

## REFERENCES

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EPA. 1996. Method 1669. Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels. U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303). Washington, DC. (July 1996).

Integral. 2004. Portland Harbor RI/FS Round 2 Field Sampling Plan, Surface Water Sampling. Prepared for the Lower Willamette Group, Portland, OR. Integral Consulting, Inc., Mercer Island, WA.

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USGS. 2000. Interagency Field Manual for the Collection of Water-Quality Data. Compiled by D.L. Lurry and C.M. Kolbe. U.S. Geological Survey, in cooperation with the U.S. Environmental Protection Agency. Open-File Report 00-213. Austin, TX.